THE MANUFACTURE OF SULPHATE OF ALUMINA AT THE COLUMBUS WATER SOFTENING AND PURIFICATION WORKS

By Charles P. Hoover[1]

A plant for making alum has recently been built and put in service at the Columbus water purification plant. This is the first plant of its kind ever built at a water purification works for making alum to coagulate water, and, although it has only been in operation a short time, it has been a success both technically and economically.

An investment of $12,000 was required for its construction, and it has been conservatively estimated that $6000 per year will be saved the city in the cost of alum. Between 800 and 1000 tons of alum will be manufactured during the coming year at a cost of about $10.50 per ton.

The process most generally employed today for coagulating and purifying water contemplates applying to the water under treatment a solution of aluminum sulphate prepared by dissolving in water the previously crystallized chemical.

In order to show the advantage of the new process, as used at Columbus, and to point out the advantage of making alum at the point where it is to be used, a brief explanation of the old process of making alum for water purification purposes will be necessary.

OLD PROCESS FOR MAKING ALUM FOR WATER PURIFICATION PURPOSES

Lump alum or sulphate of alumina is a combination of bauxite (a southern clay containing from 58 to 60 per cent alumina, the aluminum being present $Al_2O_5H_4$) with sulphuric acid (H_2SO_4). By mixing the two in lead lined tanks, and boiling for a period of from 6 to 8 hours, the first step in making alum is taken.

The following reaction takes place between the bauxite and acid.

$$Al_2O_5H_4 + 3H_2SO_4$$
$$Al_2(SO_4)_3 + 5H_2O$$

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The resultant solution is a mixture of aluminum sulphate and silica, and in order to obtain the clear aluminum solution it is necessary that the mixture be filtered.

The filtering process is perhaps the most costly, tedious and annoying step in the whole process of alum making, because the finely divided particles of silica present in crude sulphate solution quickly clog the pores of the filtering medium, and it is often necessary to force the material through the presses under considerable pressure. After being filtered the alum solution is boiled to expel the excess water. The expense of concentrating the syrup must be taken into consideration, because it is concentrated from a density of between 25° and 30° Baumé to a density of between 58° and 60° Baumé. After being concentrated the solution is discharged into trays, and, on cooling, crystallizes to the alum cake. The alum cake is then crushed or pulverized and shipped either in bulk, in barrels, or in sacks. The material, after being received at a water purification plant, is usually dissolved as needed, and a standard strength solution prepared, this solution being fed by a suitable measuring device into the water to be purified or treated.

THE HOOVER PROCESS

Bauxite and sulphuric acid are boiled in lead lined tanks until a basic solution of aluminum sulphate is obtained. The solution is then diluted with water, usually enough water is added to make 500 gallons of the solution equivalent to one ton of 17 per cent Al_2O_3 alum, and measured as needed into alum solution tanks, where it is diluted with sufficient water to make a standard solution, which is then applied to the water under treatment. By this process five distinct steps in alum making are eliminated, namely, filtering, concentrating, crystallizing, grinding and redissolving. This process is a much shortened process, being simple and inexpensive, because it consists simply in boiling bauxite with sulphuric acid and applying the resultant solution to the water under treatment.

DEPOSITION OF SLUDGE OR CHEMICAL MUD

The crude alum solution containing silica or other inert material from the bauxite, probably better defined as chemical mud, is applied to the water under treatment, the chemical mud mixes with the mud or suspended particles present in the water and finally be-

comes entrained or coagulated by the precipitated aluminum hydrate and settles out in the settling basins. The crude solution, containing the chemical mud in suspension until the metallic sulphate has been converted into hydroxide, has a function not possessed by alum solution prepared by the old process, namely, forming a matrix or nucleus for starting the coagulation, resulting not only in more efficient results with less coagulant, but also affording the process universal applicability irrespective of any lack of natural turbidity.

BAUXITE[2]

The first discovery of bauxite in America was in 1887 at a point a few miles southeast of Rome, Georgia. At present the known workable deposits of bauxite are limited to a few localities in Europe and the United States. Its occurrence in Europe is in France, Germany, Austria and Ireland; and in the United States, in Georgia, Alabama, Arkansas and New Mexico.

The mining of bauxite in the southern states has shown a great growth during the last few years. The annual production passed the 150,000 long ton mark in 1911. The production of bauxite in the United States from 1900 to 1911, inclusive, is shown in the following table.

YEAR	GEORGIA AND ALABAMA	ARKANSAS	TOTAL	VALUE
1900	19,739	3,445	23,184	$89,676
1901	18,038	867	18,905	79,914
1902	22,677	4,645	27,322	120,336
1903	22,374	25,713	48,087	171,306
1904	21,913	25,748	47,661	235.704
1905	15,173	32,956	48,129	240,292
1906	25,065	50,267	75,332	368,311
1907			97,776*	480,330
1908	14,446	37,703	52,167	263,968
1909	22,227	106,847	129,101	679,447
1910	33,096	115,836	148,932	716,258
1911	30,170	125,448	155,618	750,649

* Production of Tennessee included.

[2] From Geological Survey of Georgia, Bulletin No. 11.

Mining of bauxite

As a rule it is very easy to mine because of its comparative softness beneath the surface, it being soft enough in most cases to be dug up with a pick, although black powder is sometimes used to loosen it from its position. The bauxite is usually removed in steps or benches, and loaded into small mine cars. The ore contains a large percentage of uncombined water, which, unless expelled or driven off before shipment, makes it an important factor in the freight cost. The ore after being crushed is passed through long rotary kiln driers, similar to rotary cement kilns.

Three grades of ore are mined, first, that used in the manufacture of metallic aluminum; second, that used in the manufacture of alundum, and third, that used in the manufacture of aluminum salts.

On account of the strict specifications drawn for sulphate of alumina, by those in charge of water purification plants and paper mills, only the ore containing less than 2 per cent of ferric oxide (Fe_2O_3) is used in making aluminum sulphate. The more ferruginous ore is used in the manufacture of metallic aluminum, and the very purest grade of ore is used for the manufacture of alundum.

The following was taken from Bulletin No. 11, Geological Survey of Georgia:

> The possible yield of bauxite is greatly limited by the fact, that nearly all but the first grade material is discarded; thereby necessitating the exclusion of a vast quantity of ore, which should, by proper skill and manipulation, find ready utilization at good prices. This condition was, perhaps, made necessary in the beginning, when markets had to be established, in order that home material might compete with the cheap and less pure foreign bauxites of long standing and reputation. The principle has been so rigidly adhered to, by both operator and consumer, during the period of working in the American fields; that it has resulted in creating a demand for the first grade ore only, with practically no sales for the lower grade bauxite.

The writer believes that it is not necessary to demand bauxite of highest purity for making of aluminum sulphate to be used for water purification purposes, and it seems entirely possible that much of the cheaper grade bauxite could be used to advantage in making alum for water purification purposes.

SPECIFICATION FOR BAUXITE USED AT COLUMBUS

The specifications for bauxite now used at the Columbus plant are as follows:

Bauxite to be crushed and dried to contain not over 3 per cent moisture, the analysis to be:

Al_2O_3 not less than 52 per cent.

Fe_2O_3 not more than 3 per cent.

The contract for furnishing the supply needed for the period ending July 1, 1915, was awarded at $5.50 per ton, 2240 pounds, f.o.b. Bauxite, Arkansas. The freight rate from Bauxite, Arkansas, to Columbus, Ohio, is $4.40 per long ton, therefore, a ton of 2000 pounds delivered to Columbus, Ohio, costs $8.84.

Bauxite contains from 58 to 60 per cent Al_2O_3, whereas, ordinary filter alum usually contains 17 per cent Al_2O_3, therefore, one ton of bauxite will make a little more than three tons of alum ($Al_2(SO_4)_3$ $14H_2O$).

Bulletin No. 11, Geological Survey of Georgia, published in 1904, lists the following companies as either owning or working bauxite deposits in Georgia:

The Republic Mining and Manufacturing Company.

The Georgia Bauxite and Mining Company.

The Dixie Bauxite Company.

The Southern Bauxite Mining and Manufacturing Company.

Dealers known to the writer who are in position to quote prices on bauxite are:

Aluminum Company of America, Pittsburgh, Pa.

Mr. Winthrop C. Neilson, 1111 Harrison Building, Philadelphia, Pa.

National Bauxite Company, Philadelphia, Pa.

Globe Bauxite Company, Joliet, Ill.

HALLOYSITE

Good filter alum may also be made from halloysite. The approximate chemical analysis of which is as follows:

	Per cent
Soluble alumina	38
Insoluble (silica, etc.)	40
Water	22

Several manufacturers have attempted to make crystallized alum from halloysite, but have failed, because it has been found almost impossible to filter or separate the insoluble portions of the halloysite from the aluminum sulphate syrup. At the Columbus water purification plant, one tank, equivalent to about 6 tons of alum, has been made and used and the results obtained show that aluminum sulphate can be made from halloysite by the process being used at the Columbus water purification plant. Several carloads of this material have been purchased, and further experiments with the use of halloysite as a base for making alum solution will be made.

The North America Chemical Company, with offices at Urbana, Ohio, have a very large deposit of this ore, estimated to be 15,000,000 tons, located near Rome, Georgia, and they have assured the writer a number of times, that if a market can be created for halloysite, the material can be furnished f.o.b. cars, Rome, Georgia, at approximately $2 a ton.

SPECIFICATIONS FOR SULPHURIC ACID

Sulphuric acid, to be used for making alum, is contracted for, at the Columbus water purification works, under the following specifications:

The sulphuric acid shall be that known as 66° Baumé contact process acid, and shall contain not less than 92 per cent H_2SO_3.

The material shall be delivered at a uniform rate of not less than 30 tons per month, or at such increased rate as shall be directed.

The sulphuric acid shall be shipped in acid tank cars, provided with all necessary connections for removing the acid from the car by air pressure. Each tank shipment not to contain more than 30 tons of sulphuric acid.

Each tank car lot as a unit shall be the basis for accounting for determining of the amounts payable to the contractor.

GENERAL DESCRIPTION OF PLANT

The plant comprises: 2 lead lined boiling tanks, 2 alum measuring tanks, 1 acid measuring tank, 1 sludge tank, 1 storage tank for sulphuric acid, 1 crusher for crushing bauxite, 1 pulverizer for pulverizing bauxite, conveying, elevating and transmission machinery, bauxite storage bins and weighing device, piping, valves and fittings.

COST OF OPERATION

The following is an estimate of the cost of producing 1000 tons of 17 per cent R_2O_3 alum solution.

468 tons 66° Baumé sulphuric acid at $12.50	$5,850.00
265 gross tons bauxite at $9.90	2,623.50
Lubricating oil	20.00
Steam	100.00
Electric current 10,000 kilowatt hours (2 cents per kw. hr.)	200.00
Repairs to plant	500.00
Depreciation	600.00
Interest on investment	600.00
	$10,493.50

At the Columbus plant it is hardly fair to make any charge for labor because no additional labor is required to operate the new alum plant, consequently the labor item has been omitted in the above estimate.

When lump alum was being used here it was necessary to unload it from the cars, sack and weigh it, store it in the storage house, elevate it to the third floor of the head house, stack it up there and finally dissolve it in solution tanks as needed. In addition to all this handling there were three times as much material to move and three times as much storage space required, because, as has already been explained, 1 ton of bauxite will make 3 tons of alum. No additional help has been employed since this plant has been installed.

DISCUSSION

MR. J. M. DIVEN: How about other chemicals, iron, or possibly arsenic, in the product?

MR. CHARLES P. HOOVER: Three grades of ore are mined, first, that used in the manufacture of metallic aluminum; second, that used in the manufacture of alundum, and third, that used in the manufacture of aluminum salts. At the present time only ore containing less than 2 per cent of ferric oxide (Fe_2O_3) is used in making aluminum sulphate. The speaker believes, however, that it is not necessary to demand bauxite of highest purity for making aluminum sulphate to be used for water purification purposes. It seems entirely possible that much of the cheaper grade bauxite could be used to ad-

vantage in making alum for water purification purposes. Low grade, cheap sulphuric acid contains rather a high percentage of arsenic, and when such acid is used in the manufacture of sulphate of alumina the product will of course contain arsenic. In order to get arsenic free acid it is necessary to specify contact process acid. Professor Bartow, of the Illinois State Water Survey, read a paper at the New Orleans meeting of the American Chemical Society on the presence of arsenic in filter alum, and in this paper it was stated that the quantity of arsenic present in commercial alum was so small that even if water were treated with 6 grains per gallon of highest arsenic content alum which could be found on the market it would be necessary to drink 5000 gallons of such treated water to get a medicinal dose.

PROF. J. M. CAIRD: What is the price of sulphuric acid?

MR. CHARLES P. HOOVER: At the present time we are paying $12 per ton for sulphuric acid of 66° acid. We have a contract for a year at that price.

MR. A. W. HAWKES: The market price of sulphuric acid at the present time as compared with the price ruling at the time Mr. Hoover made his contract for sulphuric acid has practically doubled, due to the heavy demand brought about by war conditions. It might be very difficult for a water company that has not previously purchased sulphuric acid to secure any at this time at a usable price. Pyrites ore, which comes in material quantities from Spain, is used largely in this country in the production of sulphuric acid and the ocean freights have more than doubled since the outbreak of the war.

MR. CHARLES P. HOOVER: Filter alum, made by the old process, or made and used by the process just described, is made from sulphuric acid, and therefore, it naturally follows that if the price of sulphuric acid increases the selling price of alum will be increased. Even though the cost of acid does increase or double there still remains the same saving in the production of alum by this process over the production by the old process.

A MEMBER: Has the halloysite making of alum not proved successful?

MR. CHARLES P. HOOVER: Attempts to make alum from halloysite, so far as the speaker knows, have proved unsuccessful. A plant for making alum from halloysite was recently built at Urbana, Ohio, but it was only operated for a few months and has now been dismantled. It was found to be practically impossible to filter silica from the crude halloysite syrup. At the Columbus water softening and purification works, where the crude syrup is being used without filtering, concentrating or crystallizing, halloysite is being used successfully.

A MEMBER: You say that the bauxite was mixed in the tank which you illustrated. Do you boil it with a steam coil?

MR. CHARLES P. HOOVER: Briefly the process is as follows: 2 feet of water is first run into the boiling tanks, which are 8 feet in diameter, then the proper quantity of sulphuric acid is introduced. The reaction between the acid and water is so violent as to cause the solution to boil. Bauxite is then dribbled into the boiling acid solution at the rate of about 30 pounds per minute. The reaction between the hot acid solution and the bauxite is violent and the boiling continues as long as the bauxite is being introduced, this usually takes about two hours' time. Live steam is then used to continue the boiling process to complete the reaction between the alumina, of the bauxite, and the sulphuric acid.

PROF. JAMES M. CAIRD: How much of that available alumina is consumed by the sludge? How much of it is lost in the sludge? Did you not say about 40 per cent?

MR. CHARLES P. HOOVER: No; about 6 or 7 per cent of the available alumina is lost.

MR. PHILIP BURGESS: What has been the attitude of the filter alum manufacturers in regard to this process? Have they intimated that in view of the experience at Columbus it might be practical and desirable for them, instead of furnishing the pure crystallized chemical, to contemplate a supply of the materials in the liquid form?

MR. CHARLES P. HOOVER: Will Mr. Hawkes, of the General Chemical Company, kindly answer the question which has just been asked by Mr. Burgess?

MR. A. W. HAWKES: It would not be feasible or practicable for an alum manufacturer to attempt to transport alum solution for the reason that this solution has an acid reaction and attacks most metals, hence it would require a very expensive vessel in which to ship it. Then, too, there would be the question of paying freight from point of shipment to destination on the water in the alum solution.

From the speaker's past experience, he does not believe the manufacturers of alum will make any material advance in the price of sulphate of alumina to their regular customers, no matter where the price of sulphuric acid goes, as it has always been the policy of the manufacturers in the past to take care of their American trade, and contracts are being renewed now from day to day, regardless of the price of sulphuric acid, on the same price basis as ruled before the war. Generally speaking, it is believed the policy of the American alum manufacturer will be to take care of its regular customers in this country on practically the same basis they have been taken care of in the past.